

N85-32413

A FLUIDIZED-BED REACTOR FOR SILANE PYROLYSIS

UNION CARBIDE CORP.

S. Iya

<u>TECHNOLOGY</u> POLYCRYSTALLINE SILICON R&D	<u>REPORT DATE</u> OCTOBER 2, 1984
<u>APPROACH</u> SILANE DECOMPOSITION IN A FLUIDIZED BED REACTOR <u>CONTRACTOR</u> UNION CARBIDE CORPORATION	<u>STATUS</u> <ul style="list-style-type: none">● HIGH-PURITY LINER WAS INSTALLED IN THE FLUID BED REACTOR.● LONG DURATION TEST RUNS WERE CONDUCTED.● FBR PRODUCT WAS MELTED AND SINGLE CRYSTALLIZED.● PRODUCT PURITY IMPROVEMENTS WERE NOTED.
<u>GOALS</u> <ul style="list-style-type: none">● DEMONSTRATE PROCESS FEASIBILITY.● DETERMINE OPERATING WINDOW.● CONDUCT LONG-DURATION TESTS.● DEMONSTRATE SILICON PURITY.	

Summary of Activities

- PDU WAS MODIFIED TO INSTALL A HIGH-PURITY LINER.
- A SUITABLE LINER SUPPORT SYSTEM WAS DESIGNED AND IMPLEMENTED.
- SEED BED WAS PREPARED BY SCREENING AND ACID WASHING PURCHASED SILICON FINES.
- A LONG-DURATION TEST RUN WAS CONDUCTED USING POLYSILICON LINER. PRODUCT FROM THIS RUN WAS SINGLE CRYSTALLIZED AND ANALYZED FOR PURITY.
- A HIGH-THROUGHPUT TEST RUN WAS CONDUCTED USING QUARTZ LINER.
- A COLD MODEL WAS CONSTRUCTED TO INVESTIGATE COARSE PARTICLE WITHDRAWAL.

SILICON MATERIAL

Run Summary: Long-Duration Test With Polysilicon Liner

- 56 HOURS RUN DURATION FOLLOWED BY VOLUNTARY SHUTDOWN.
- 280 μ M SEED GROWN TO 500 μ M PRODUCT.
- SILANE FEED CONCENTRATION IN THE RANGE 10 - 15%.
- AVERAGE DEPOSITION RATE APPROXIMATELY 1 KG/HR.
- BED TEMPERATURE 650 - 750°C.
- U/UMF 3.5 - 4.0.
- COMPLETE SILANE CONVERSION WITHIN THE BED.
- SEVERAL KG PRODUCT WAS WITHDRAWN.
- FINE POWDER 5.3 % OF SILANE FEED.
- POWER CONSUMPTION 25 KWH/KG.

Long-Duration Run: Mass Balance

INITIAL BED WEIGHT	=	26.7 KG
SILICON IN	=	63.0 KG
TOTAL	=	<u>89.7 KG</u>
BED MATERIAL WITHDRAWN	=	83.5 KG
POWDER IN FILTER HOPPERS	=	<u>3.8 KG</u>
TOTAL	=	87.3 KG
ERROR IN MASS BALANCE	=	2.7%

Run Summary: High Throughput Test With Quartz Liner

- 10 HOURS TOTAL RUN DURATION.
- MAXIMUM SILANE FEED CONCENTRATION 48%
- MAXIMUM DEPOSITION RATE 3.8 KG/HR.
- FINE POWDER 6.9% OF SILANE FEED
- POWER CONSUMPTION 8 KWH/KG.
- SHUT DOWN CAUSED BY HEATER FAILURE.

Test Product Characterization:
Long-Duration Run With Poly Liner

PARTICLE PROPERTIES

- 500 μM MEAN PARTICLE DIAMETER
- 100 LB/CFT. BULK DENSITY
- SMOOTH, ROUNDED SURFACE
- FREE FLOWING

PARTICLE MORPHOLOGY

- DENSE DEPOSITION LAYER
- LAYERED RING-LIKE GROWTH STRUCTURE
- GROWTH LAYER THICKNESS $\sim 100 \mu\text{M}$

PARTICLE PURITY

- FE, CR, NI NOT DETECTED BY EMISSION SPEC
(FE < 20 PPM, CR < 5 PPM; NI < 5 PPM)
- SAMPLES OF SEED, INTERMEDIATE & FINAL PRODUCT
WERE SENT TO JPL FOR NEUTRON ACTIVATION ANALYSIS
- SINGLE CRYSTAL RESISTIVITY 8 OHM-CM, P TYPE
- FTIR MEASUREMENTS SHOWED PPB LEVELS OF BORON AND
PHOSPHOROUS

Plans

- ADDITIONAL PURITY RUNS STARTING WITH UNION CARBIDE
SEED MATERIAL.
- PRODUCT PURITY EVALUATION.
- COARSE PRODUCT WITHDRAWAL TESTS.
- TECHNICAL AND ECONOMIC ASSESSMENT.